

# Fate of Endosulfan in Cotton Soil under Sub-tropical Conditions of Northern India\*

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**Abstract:** Terrestrial field studies were conducted with endosulfan during the 1989–90 *Kharif* season in bare cotton soil, to investigate the fate of endosulfan and its downward movement under sub-tropical conditions of northern India. Field experiments consisted of spray application of endosulfan at 875 g ha<sup>-1</sup> 42 and 63 days after the assumed date of sowing in two separate treatments. Soil samples drawn periodically from different depths were analysed by GC-ECD (Ni<sup>63</sup>) for endosulfan and its breakdown products.

Dissipation of the total endosulfan residues occurred to an extent of 92–97% in the first four-week period and by about 99% in 238 days in two distinct phases in first-order kinetics. Residue half life ( $T_{1/2}$ ) varied from 39 to 42 days. The parent compound metabolized to endosulfan-diol and endosulfan sulfate. Endosulfan-diol remained confined in the upper 5-cm layer and dissipated completely in 28 days whereas endosulfan sulfate was first detectable seven days after treatment and persisted until the end of the experiment, remaining confined in the upper 0–10 cm soil layer. The  $\beta$ -isomer also did not leach down beyond 10 cm depth.

Key words: endosulfan, dissipation, persistence, metabolites, residue half life ( $T_{1/2}$ ), sub-tropical conditions

## 1 INTRODUCTION

For about the last three decades endosulfan [(1, 4, 5, 6, 7, 7-hexachloro-8, 9, 10-trinorborn-5-en-2, 3-ylenebis-methylene)sulfite] has been widely used for controlling different insect pests of field crops such as paddy, cotton, sorghum, oilseeds, pulses, vegetables and fruit crops under sub-tropical conditions of India. In the cotton-growing belt of northern India the compound is recommended for controlling hairy caterpillar, spotted bollworm, semi-looper, etc. on cotton.<sup>1</sup> In recent years, it has been tested for controlling termites in soil as an alternative to aldrin, which has been withdrawn from the Indian market since December 1993 due to its high persistence.

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Soil serves as a major sink for plant protection chemicals applied directly to soil or entering indirectly during plant protection operations or through crop residues of treated plants. Xenobiotics present in soil may be absorbed by plants or may dissipate by chemical, microbial degradation, downward leaching or volatilization. Additionally, some portion of the chemical may become part of soil system in the form of bound residues. The dissipation pattern of endosulfan has been extensively documented on different crops<sup>2–6</sup> under sub-tropical conditions but very little information is available on its fate and dissipation behaviour in cotton soils. Under northern Indian conditions, dissipation of endosulfan residues was observed to be about 63% in two months under cover of sorghum in alluvial soils.<sup>3</sup> The sandy loam character of the cotton soil enhances the chances for downward movement of pesticides which may result in the contamination of ground water. It was thought imperative to undertake investigations

to study the fate, movement and dissipation of endosulfan in cotton soil without a growing crop under sub-tropical condition of Hisar, India, focusing on its potential for contaminating ground water.

## 2 MATERIALS AND METHODS

### 2.1 Chemicals

Endosulfan 357 g litre<sup>-1</sup> EC ('Thiodan' 35) and reference standards of endosulfan isomers, endosulfan-diol and sulfate were obtained through courtesy of Hoechst Schering AgrEvo Ltd, Bombay; the derivatizing reagent, *N*-methyl-*N*-trimethylsilyl-trifluoroacetamide (MSTFA) was procured by courtesy of M/s Hoechst Schering AgrEvo Ltd, Frankfurt, Germany. All reagents used for extraction, clean-up and other purposes were of analytical grade.

### 2.2 Field metabolism and movement experiment

The field experiment was conducted during May 1989–April 1990 at the Research Farm of CCS Haryana Agricultural University, Hisar, India. The soil was sandy loam having 67.3% sand, 13.8% silt, 18.6% clay, pH measured with KCl (1:2) 8.10; CEC [c mol (P + t) kg<sup>-1</sup>] 11.45; C + N, 11 + 1; organic matter 0.5%; saturation, 33.40% and bulk density 1.46 Mg m<sup>-3</sup>. Out of 0.40 ha area prepared for field studies, three plots each measuring 500 m<sup>2</sup> were demarcated for three treatments (T), leaving a border area of 2.5 to 6 m around the experimental plots. In the centre of each plot an area of 15 × 15 m was marked as sampling area.

In T<sub>1</sub> and T<sub>2</sub> endosulfan EC was applied at 875 g AI ha<sup>-1</sup> in 375 litre water 42 and 63 days after the assumed date of sowing, i.e. 21 June 1989; T<sub>3</sub> was kept as the untreated control plot. To ensure uniform coverage of the field endosulfan emulsion was sprayed in cross directions with a knapsack sprayer.

Throughout the experiment, the plots were kept free of weeds by hand hoeing, taking care not to disturb the upper layer of soil. The plots were irrigated six times. The first irrigation was given 28 days after the assumed date of sowing at 100 litre m<sup>-2</sup>, second at 75 litre m<sup>-2</sup> and thereafter at 50 litre m<sup>-2</sup> at a 21-day intervals. This schedule was selected as if irrigation would have been carried out under normal cropping conditions. On the assumed date of sowing 30 kg ha<sup>-1</sup> each of nitrogen and P<sub>2</sub>O<sub>5</sub> were applied during field preparations. An additional 30 kg ha<sup>-1</sup> of nitrogen was given prior to the second irrigation.

In order to observe the fate and downward movement of endosulfan and its metabolites, soil samples were drawn at eight time intervals (–1 to 238 days) at four different depths (Table 1). Soil samples were drawn

**TABLE 1**  
Sampling Schedule Followed for T<sub>1</sub> and T<sub>2</sub>

Sampling interval (DAT) <sup>a</sup>	Depth of sampling <sup>b</sup>
–1	d <sub>1</sub> , d <sub>2</sub> , d <sub>3</sub> , d <sub>4</sub>
0	d <sub>1</sub>
7	d <sub>1</sub>
14	d <sub>1</sub> , d <sub>2</sub>
28	d <sub>1</sub> , d <sub>2</sub> , d <sub>3</sub>
70	d <sub>1</sub> , d <sub>2</sub> , d <sub>3</sub> , d <sub>4</sub>
154	d <sub>1</sub> , d <sub>2</sub> , d <sub>3</sub> , d <sub>4</sub>
238	d <sub>1</sub> , d <sub>2</sub> , d <sub>3</sub> , d <sub>4</sub>

<sup>a</sup> DAT = Days after treatment.

<sup>b</sup> d<sub>1</sub> = 0–5 cm; d<sub>2</sub> = 5–10 cm; d<sub>3</sub> = 10–15 cm; d<sub>4</sub> = 15–30 cm.

randomly with the help of a tube auger (dia 2.5 cm) on each date from 40 different places in a marked 15 × 15 m area. All core samples of different depths were mixed separately and, after quartering, two composite samples were drawn from each depth and treatment. Samples were packed in polyethylene boxes lined with aluminium foil and stored in a deep freeze at a temperature below –30°C until the analysis, which was carried out within one to three months of sampling.

Meteorological conditions except rainfall prevailing during the experiment were quite normal compared to the climatic conditions of the last ten years. The maximum, minimum temperatures, pan evaporation rate and rainfall during the experimental period as well as long-term values are depicted in Fig. 1.

### 2.3 Extraction and clean-up

Soil samples were air dried, passed through a 2-mm sieve and quartered to obtain a representative 50-g sample after homogenization. Acetone extraction was carried out according to the method of Werner *et al.*<sup>7</sup> The acetone extract was diluted with aqueous sodium chloride solution (40 g litre<sup>-1</sup>; 600 ml) and residues partitioned to hexane (3 × 50 ml). The combined hexane phases were evaporated to about 35 ml on a rotary flash evaporator at 40–45°C. The hexane extract was cleaned up with an improved technique<sup>8</sup> using Darco-G-60 as adsorbent and hexane + toluene (95 + 5 by volume) as eluant. The eluates collected were evaporated to a volume of 10 ml on a flash evaporator.

### 2.4 Derivatization of endosulfan and its metabolites

From the cleaned extract (10 ml) an aliquot (0.02 ml) was taken in a graduated injection bottle and diluted to 0.1 ml with toluene. The silylation<sup>7</sup> was done with 0.04 ml MSTFA in a capped injection bottle by heating at 70–80°C for 15 min. After cooling of solution toluene was added up to a final volume of 1 ml.

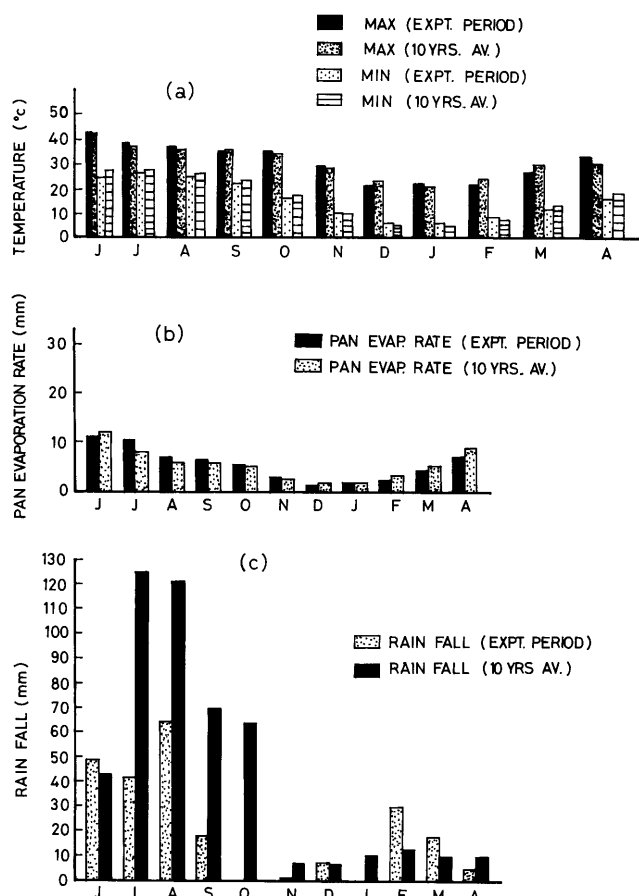


Fig. 1. Meteorological observations at the experimental site: (a) air temperature (b) pan evaporation rate and (c) rainfall (monthly averages) of experimental period (June 1989 to April 1990) and of last 10 years.

The derivatized residues were determined by injecting 1–3  $\mu$ l of toluene solution into a gas liquid chromatograph (GLC, HP 5890A), equipped with  $\text{Ni}^{63}$  detector and a HP-1 (methyl silicone gum) 10 m  $\times$  0.53 mm  $\times$  2.65  $\mu$ m film thickness column. The injection port and detector temperatures were 265°C and 270°C, respectively. The column temperature was set to 200°C (1 min)—3°C min<sup>-1</sup> to 215°C (2 min)—20°C min<sup>-1</sup> to 260°C (2 min). The gas flow ( $\text{N}_2$ ) was set to 15 ml through column and 55 ml make-up through detector. The retention times ( $R_t$ ) observed under these conditions were: endosulfan- $\alpha$ , 3.20 min; endosulfan  $\beta$ ; 4.03 min; endosulfan sulfate, 4.95 min, endosulfan diol 3.75 min.

## 2.5 Recovery experiments

Before starting the analysis of test samples, recovery experiments were conducted with soil samples from the control plot fortified at levels of 0.1 and 0.5 mg kg<sup>-1</sup>. Each fortification level was prepared in three replicates. Recoveries observed for  $\alpha$ - and  $\beta$ -isomers of endosulfan,

endosulfan sulfate and endosulfan-diol were 98%, 106%, 95% and 95%, respectively.

The limits of detection were 0.01  $\mu$ g g<sup>-1</sup> for each of the endosulfan isomers and breakdown products.

## 3 RESULTS AND DISCUSSION

### 3.1 Environmental fate and persistence behaviour of endosulfan in soil

In both treatments, endosulfan was detected in the form of  $\alpha$ - and  $\beta$ -isomers on the day of application (Tables 2, 3). In seven days endosulfan was converted to endosulfan sulfate in  $T_1$  as well as in  $T_2$ . The formation of endosulfan-diol was observed in  $T_1$  only.

The endosulfan  $\alpha$ -isomer persisted up to 14 days in  $T_1$  and 28 days in  $T_2$ . The endosulfan  $\beta$ -isomer was relatively more persistent than the  $\alpha$ -isomer. Its residues persisted up to 70 days in  $T_1$  and 238 days in  $T_2$  in detectable amounts. These results confirm observations of Kimber *et al.*<sup>9</sup> with endosulfan in cotton soils of Australia, that, although the endosulfan  $\alpha$ -isomer is 70% of the active ingredient in commercial formulations, because of its high volatility it is found in soils at appreciable levels only immediately after spraying.

The endosulfan-diol persisted up to 14 days. The endosulfan sulfate residues in the soil samples were highest on day 7 in  $T_1$  and day 14 in  $T_2$  with 0.666 mg kg<sup>-1</sup> and 0.143 mg kg<sup>-1</sup>, respectively. Endosulfan sulfate persisted until the termination of the experiment in both treatments, always being around four times higher in  $T_1$  than in  $T_2$  (Tables 2 & 3). This observation is in accordance with findings on environmental fate of endosulfan in Australian cotton soils,<sup>9</sup> where also endosulfan sulfate was the main breakdown product followed by endosulfan-diol.

Dissipation of endosulfan occurred in two phases in first-order kinetics (Table 4; Fig. 2). Residue half-life ( $T_{1/2}$ ) values varied from 5.4 to 7.3 days in the first phase, 78.6 to 115.3 days in the second phase and from 39.5 to 42.1 days for overall period. Residue half-life of 15 days for endosulfan has been reported in Australian black soil<sup>10</sup> when incubated at 30°C at field capacity moisture level.

In the first phase (28 days after treatment) the rate of dissipation was slightly faster in  $T_1$  (32–95%) than  $T_2$  (27–92%) whereas in the second phase (28–238 days) the rate of dissipation was similar in both treatments.

### 3.2 Movement of endosulfan and its metabolites

As evident from the data given in Tables 2 and 3 endosulfan and its breakdown products did not move beyond 10 cm depth (Fig. 3).  $\alpha$ -Endosulfan remained confined in 0–5 cm in  $T_1$  but moved down to 10 cm in  $T_2$ . The  $\beta$ -endosulfan leached down to 10 cm, both in

**TABLE 2**  
Dissipation of Endosulfan in Cotton Soil in T<sub>1</sub> (Treatment given 42 Days after Assumed Date of Sowing)

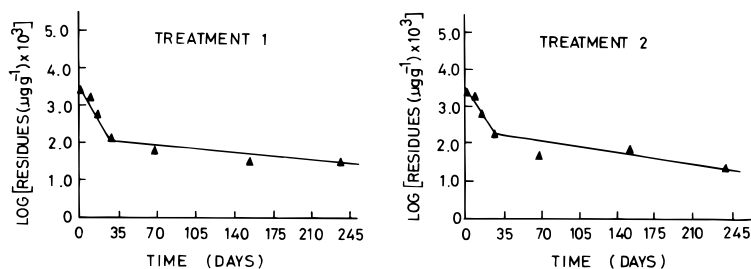
DAT	Depth (cm)	Endosulfan residues (mg kg <sup>-1</sup> ) <sup>a</sup>					Dissipation (%)
		$\alpha$ -isomer	$\beta$ -isomer	Endodiol	Endosulfate	$\Sigma$ Endosulfan	
-1	0-30	BDL <sup>b</sup>	BDL	BDL	BDL	BDL	—
0 (1 h)	0-5	2.350	0.676	BDL	BDL	3.026	—
7	0-5	0.996	0.273	0.134	0.666	2.069	31.62
14	0-5	0.243	0.129	0.096	0.107	0.575	—
	5-10	BDL	0.036	BDL	BDL	0.036	
						0.611	79.81
28	0-5	BDL	0.034	BDL	0.109	0.143	
	5-10	BDL	0.002	BDL	BDL	0.002	
	10-15	BDL	BDL	BDL	BDL	BDL	
						0.145	95.21
70	0-5	BDL	0.009	BDL	0.058	0.067	
	5-10	BDL	BDL	BDL	BDL	BDL	
	10-15	BDL	BDL	BDL	BDL	BDL	
	15-30	BDL	BDL	BDL	BDL	BDL	
						0.067	97.78
154	0-5	BDL	BDL	BDL	0.040	0.040	
	5-10	BDL	BDL	BDL	BDL	BDL	
	10-15	BDL	BDL	BDL	BDL	BDL	
	15-30	BDL	BDL	BDL	BDL	BDL	
						0.040	98.68
238	0-5	BDL	BDL	BDL	0.034	0.034	
	5-10	BDL	BDL	BDL	BDL	BDL	
	10-15	BDL	BDL	BDL	BDL	BDL	
	15-30	BDL	BDL	BDL	BDL	BDL	
						0.034	98.88

<sup>a</sup> Mean of two replicates.

<sup>b</sup> BDL = Below detectable level.

T<sub>1</sub> and T<sub>2</sub> until 28 days of experimentation. The amount present in the 5-10 cm layer was significantly lower than in the 0-5 cm layer. Endosulfan-diol, which was detected only in T<sub>1</sub>, did not leach beyond 5 cm. Likewise endosulfan sulfate mainly remained confined in upper 5-cm layer up to the termination of the experiment except on day 154 in T<sub>2</sub> when residues to a level of 0.10 mg kg<sup>-1</sup> were found in 5-10 cm depth. Similar observations have been made by Kimber *et al.*<sup>9</sup> while

working on the environmental fate of endosulfan sprayed on cotton. They found that the bulk of the pesticide remained in top 5-cm layer in cotton-growing soils of Australia. Probably adsorption of endosulfan and its metabolites was strong on soil particles due to their polar character, resulting in negligible downward movement with irrigation and rain. Also low water solubility can be another reason for negligible downward mobility.



**Fig. 2.** Dissipation of endosulfan applied at 875 g AI ha<sup>-1</sup> in cotton soil.

	$T_{1/2}$ values	
	$T_1$	$T_2$
Phase I	5.4 DAYS	7.3 DAYS
Phase II	115.3 DAYS	78.3 DAYS
Overall period	42.1 DAYS	39.5 DAYS

**TABLE 3**  
Dissipation of Endosulfan in Cotton Soil in T<sub>2</sub> (Treatment given 63 Days after Assumed Date of Sowing)

DAT	Depth (cm)	Endosulfan residues (mg kg <sup>-1</sup> ) <sup>a</sup>					Dissipation (%)
		$\alpha$ -isomer	$\beta$ -isomer	Endodiol	Endosulfate	$\Sigma$ Endosulfan	
–1	0–30	BDL <sup>b</sup>	BDL	BDL	BDL	BDL	—
0 (1 h)	0–5	2.225	0.575	BDL	BDL	2.800	—
7	0–5	1.409	0.500	BDL	0.125	2.034	27.36
14	0–5	0.490	0.124	BDL	0.143	0.757	
	5–10	0.081	0.032	BDL	BDL	0.113	
						0.870	68.93
28	0–5	0.042	0.070	BDL	0.104	0.216	
	5–10	BDL	BDL	BDL	BDL	BDL	
	10–15	BDL	BDL	BDL	BDL	BDL	
						0.216	92.29
70	0–5	BDL	0.014	BDL	0.057	0.071	
	5–10	BDL	BDL	BDL	BDL	BDL	
	10–15	BDL	BDL	BDL	BDL	BDL	
	15–30	BDL	BDL	BDL	BDL	BDL	
						0.071	97.46
154	0–5	BDL	0.027	BDL	0.048	0.075	
	5–10	BDL	BDL	BDL	0.010	0.010	
	10–15	BDL	BDL	BDL	BDL	BDL	
	15–30	BDL	BDL	BDL	BDL	BDL	
						0.085	96.96
238	0–5	BDL	0.014	BDL	0.009	0.023	
	5–10	BDL	BDL	BDL	BDL	BDL	
	10–15	BDL	BDL	BDL	BDL	BDL	
	15–30	BDL	BDL	BDL	BDL	BDL	
						0.023	99.18

<sup>a</sup> Mean of two replicates.

<sup>b</sup> BDL = Below detectable level.

Low vertical mobility of four organochlorine insecticides, i.e., aldrin, HCH, chlordane and heptachlor was demonstrated in sandy loam soil in a long-term experiment covering ten crop seasons.<sup>11</sup> It was noticed that highest residue concentrations were present in the

surface (0–10 cm) of the fallow plots but in the 10–20 cm layer of the cropped soils.

There are many variables such as fertilizer application, irrigation, cropping pattern and other agronomical practices, which influence dissipation of pesticides under

**TABLE 4**  
First-Order Kinetics of Endosulfan Dissipation in Cotton Soil

Phase <sup>a</sup>	Regression equation <sup>b</sup>	Correlation coefficient (r)	T <sub>1/2</sub> (days)	Rate constant (K)
Treatment 1				
First	Y = 3.58 – 0.056 X	–0.99	5.4	0.129
Second	Y = 2.11 – 0.003 X	–0.91	115.3	0.006
Overall	Y = 2.92 – 0.007 X	–0.81	42.1	0.016
Treatment 2				
First	Y = 3.51 – 0.041 X	–0.99	7.3	0.095
Second	Y = 2.34 – 0.004 X	–0.89	78.6	0.009
Overall	Y = 3.009 – 0.008 X	–0.87	39.6	0.016

<sup>a</sup> First phase: 0–28 DAT; second phase: 28–238 DAT.

<sup>b</sup> Y = Log [Residues × 10<sup>3</sup>]; X = Interval (days) after treatment.

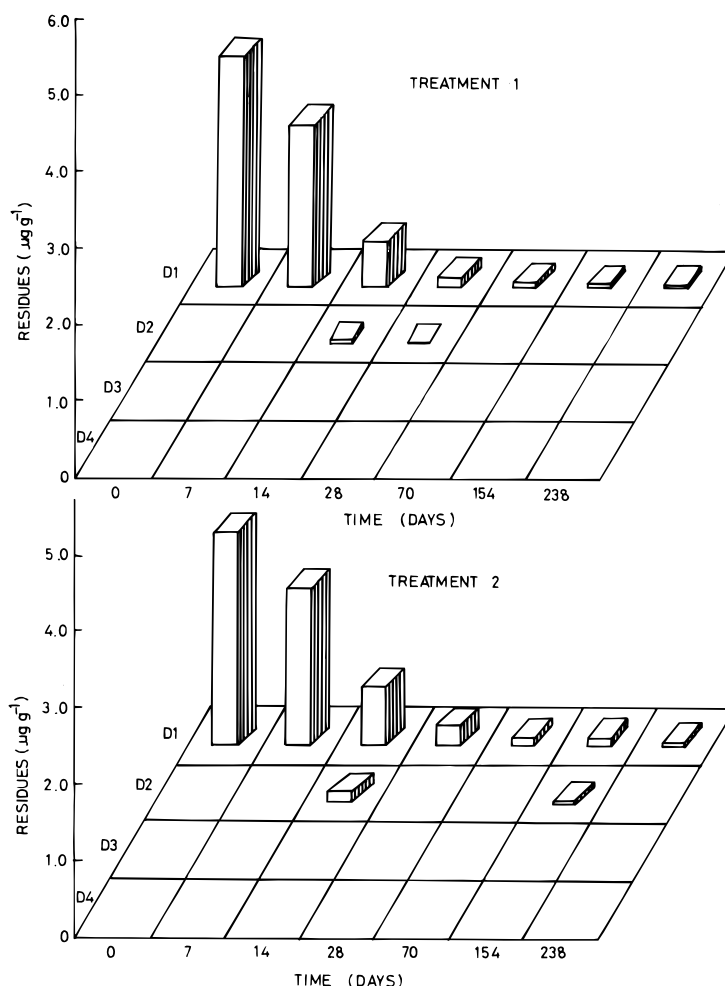


Fig. 3. Downward movement in soil of endosulfan applied at  $875 \text{ g AI ha}^{-1}$  42 days ( $T_1$ ) and 63 days ( $T_2$ ) after assumed date of sowing.  $D_1 = 0-5 \text{ cm}^{-1}$ ;  $D_2 = 5-10 \text{ cm}$ ;  $D_3 = 10-15 \text{ cm}$ ;  $D_4 = 15-30 \text{ cm}$ .

field conditions. In the current studies, the following processes can be considered to have operated in the dissipation of endosulfan (1) volatilization due to fallow plots (2) hydrolysis of endosulfan and metabolization because of alkaline pH (8.1) of soil and irrigation water (8.2) (3) photochemical and microbial decomposition. According to various researchers<sup>12,13</sup> microbial and chemical degradation are minor routes of dissipation of pesticides from soil but most are lost in significant amounts through volatilization.<sup>14,15</sup> In the light of these findings, in our studies volatilization was probably the dominant factor in the dissipation of endosulfan and its metabolites which was probably favoured by a relatively high temperature coupled with low rainfall as compared to the ten-year average values, particularly in the post-application stages both in  $T_1$  and  $T_2$  (Fig. 1). From these investigations, the conclusions can be drawn that under sub-tropical conditions (i) endosulfan was converted to endosulfan sulfate and diol in sandy loam soil (ii) endosulfan sulfate persisted in measurable amounts up to 238 days whereas diol dissipated almost completely within 28 days (iii) major quantities of resi-

dues remained confined in the upper (0–5 cm) layer; however, very small amounts moved down to 10 cm depth, (iv) dissipation of total endosulfan was fairly rapid. Hence endosulfan can be considered an environmentally sound insecticide for the use in cotton under sub-tropical conditions of northern India.

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